STRUCTURAL AND MAGNETIC CHARACTERIZATION OF COPPER MANGANESE FERRITE NANOPARTICLES SYNTHESIZED BY CHEMICAL COPRECIPITATION ROUTE

G. Vasuki¹ and T. Balu²

Abstract-The mixed spinel ferrite Cu0.5Mn0.5Fe2O4 have been synthesized by chemical coprecipitation technique. The synthesized sample has been characterized for structural, compositional and magnetic studies through SEM, EDX, powder XRD and VSM (vibrating sample magnetometer) analysis. The SEM image shows crystalline morphology. The single phase crystalline cubic spinel structure, the d spacing, lattice constant and X-ray density have been determined from the powder XRD. The average particle size using Debye Scherrer formula is calculated to be 30 nm. The VSM measures the magnetization, coercivity and retentivity values from the hysteresis. The small values of coercivity and squareness ratio shows the isotropic nature of the material.

Keywords: chemical coprecipitation, morphology, lattice constant, coercivity, isotropic.

INTRODUCTION
The nanoferrite materials find their applications in various fields of bioscience and technology. Their importance in drug delivery system, electromagnetic wave absorption, magnetic resonance imaging, induction cores, signal transmission, gas sensors have attracted many researchers to synthesis these magnetic materials[1-3]. Numerous methods have been employed for the synthesis of the nanoferrites. The combustion method, solgel process, solvothermal process, chemical coprecipitation etc... are commonly used methods. Of these the chemical coprecipitation technique is a simplest, low cost, efficient method[4,5]. In recent years many researchers have interested in synthesizing mixed spinel ferrites for improved applications[6,7]. The efficient usage of copper and manganese ferrites in biomedicine, microwave absorption and sensor devices a work has been done to synthesis copper manganese mixed spinel ferrite nanoparticles (Cu0.5Mn0.5Fe2O4) by the coprecipitation route[8,9]. The synthesised material has been characterized for powder XRD, SEM, EDX and VSM studies.

EXPERIMENTAL PROCEDURE
The high purity AR grade Merck products of sulphate salts, sodium hydroxide and demineralised water were used for the synthesis. The M²⁺:Fe³⁺ ratio was taken as 1:2. 50ml aqueous solutions of the salts were prepared as the starting solution with pH ~ 3. 50ml of 3M NaOH was used as the precipitant. The precipitant was added dropwise to the light green coloured starting solution to form a dark green intermediate precipitate. The final pH attained was 11. The obtained precipitate was heated to 70°C on continuous stirring for 2 hrs. The precipitate changes to dark brown colour. Then it was washed for about 5 times to remove the sulphates, filtered and dried at 105°C. The reaction mechanism is shown in figure 1. The dried product was powdered to obtain Cu0.5Mn0.5Fe2O4 nanoparticles.

50 ml starting solution + 50 ml precipitant \[\Delta 70^\circ C\] intermediate precipitate

Nanopowders \[\text{washed, filtered, dried}\] final precipitate

Figure 1: reaction mechanism for synthesis of Cu0.5Mn0.5Fe2O4 nanoparticles.

The obtained nanoparticles were analyzed for crystalline structure using powder X-ray diffractometer(pXRD), morphological study using Scanning Electron Microscopy (SEM), compositional analysis by energy dispersive analysis of X-ray (EDX) and the magnetic behaviour at 300K using Vibrating Sample Magnetometer(VSM).

RESULTS AND DISCUSSIONS
The crystal structure and phase of the synthesized product Cu0.5Mn0.5Fe2O4 was analyzed using an X-ray diffractometer(XPERT-PRO model) utilizing CuKα radiation source of \(\lambda=1.5406\ A\) (figure 2). The formation of a single phase cubic spinel ferrite structure of the powders was confirmed. The reflections at various 2θ’s corresponds to the miller indices (220), (311), (400), (511), (440) as per the JCPDS card no 19-0629 for magnetite[10].

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Structural And Magnetic Characterization Of Copper Manganese Ferrite Nanoparticles Synthesized By Chemical Coprecipitation Route

From the maximum intensity peak the average crystallite size was determined using Debye Scherrer formula [11]: 

\[
D = \frac{0.9 \lambda}{\beta \cos \theta}
\]

where \(\beta\) is the full width at half maximum of the peak at a particular diffraction angle and \(\lambda\) is the wavelength of the source used. The estimated particle size is about 30 nm. The \(d\) spacing, lattice constant, X-ray density corresponding to the (311) plane is given in table 1. The lattice constant value was calculated using the following formula[12]:

\[
a = d\sqrt{h^2+k^2+l^2}
\]

where \(d\) is the interplanar spacing and \(h,k,l\) are the miller indices. The lattice constant value obtained is lower than that of the bulk material [12]. The X-ray density is calculated using the formula [13]:

\[
\rho = 8M/Na^3
\]

where \(M\) is the molecular weight of the sample and \(N\) is the Avagadro number.

Table 1: Values determined from pXRD pattern

<table>
<thead>
<tr>
<th>Miller index</th>
<th>(d) spacing (Å)</th>
<th>Lattice constant (a) (Å)</th>
<th>X-ray density (\rho) (g/cm(^3))</th>
</tr>
</thead>
<tbody>
<tr>
<td>311</td>
<td>2.5407</td>
<td>8.4196</td>
<td>5.239</td>
</tr>
</tbody>
</table>

The morphological analysis was performed using SEM. Figure 3 shows the SEM image of \(\text{Cu}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4\) nanopowders. It can be seen from the image that a rod like morphology of the particles. They show agglomerated, well defined nanoparticles. The agglomeration of particles is due to the permanent magnetic moment possessed by the particle[14].

The EDX analysis of \(\text{Cu}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4\) was taken to identify the elemental composition. The pattern indicates the presence of copper, manganese, iron, oxygen and traces of sulphur in the ferrite samples.

The magnetization curve of the synthesized samples obtained using vibrating sample magnetometer analysis.
Figure 5: The magnetization curve of as prepared Cu$_{0.5}$Mn$_{0.5}$Fe$_2$O$_4$ nanopowders. The magnetic moment per unit cell $M_B$ was calculated from magnetisation data using the equation [15]:

$$M_B = \text{molecular weight x saturation magnetisation density x avogadro number x } \mu_B$$

The values of the saturation magnetisation $M_s$, retentivity $M_r$, the coercivity $H_c$ and the magnetic moment $M_B$ obtained from the hysteresis curve are given in Table 2.

**Table 2: Parameters determined from magnetic studies**

<table>
<thead>
<tr>
<th>$M_s$ (emu)</th>
<th>$M_r$ (x10$^{-3}$ emu)</th>
<th>$H_c$ (G)</th>
<th>$M_B$ (μB)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.85773</td>
<td>67.834</td>
<td>60.812</td>
<td>5.56</td>
</tr>
</tbody>
</table>

The squareness ratio ($M_r/M_s$) indicates the direction with which the magnetization reorients after the magnetic field is removed. The small value of squareness ratio and coercivity shows the isotropic nature of the material [16]. This property of the material finds its usage in biomedicine and as an absorbing material.

**CONCLUSION**

The mixed spinel copper manganese nanoferrites was successfully synthesized by coprecipitation route. From the pXRD pattern the sample shows a single phase cubic spinel structure in good agreement with JCPDS card number with a particle size in the nanorange. The SEM micrograph shows fine crystalline rod like morphology and the composition from EDX shows the presence of exact cations. The hysteresis curve from VSM analysis shows a low value of coercivity and squareness ratio, an essential need for the isotropic behaviour and also leads to the superparamagnetic behaviour for extended applications.

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**REFERENCES**


List of figures

- Figure 1: Reaction mechanism for synthesis of Cu$_{0.5}$Mn$_{0.5}$Fe$_2$O$_4$ nanopowders.
- Figure 2: Powder XRD spectra of as prepared Cu$_{0.5}$Mn$_{0.5}$Fe$_2$O$_4$ nano powders.
- Figure 3: SEM micrograph of as prepared Cu$_{0.5}$Mn$_{0.5}$Fe$_2$O$_4$ nano powders.
- Figure 4: EDX pattern of as prepared Cu$_{0.5}$Mn$_{0.5}$Fe$_2$O$_4$ nano powders.
- Figure 5: The magnetization curve of as prepared Cu$_{0.5}$Mn$_{0.5}$Fe$_2$O$_4$ nano powders.

List of tables

- Table 1: Values determined from pXRD pattern.
- Table 2: Parameters determined from magnetic studies.